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EXPERIMENTAL INVESTIGATION OF GLASS FLAKES AS A LINER FOR FIBER-GLASS CRYOGENIC PROPELLANT TANKS

by Robert W. Frischmuth, Jr.

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SUMMARY

The use of filament-wound fiber-glass liquid-hydrogen propellant tank structures should result in a considerable weight savings providing that a suitable internal liner can be developed. This report investigates a liner made of a matrix of glass flakes and epoxy resin bonded to the inner surface of a filament-wound shell. A possible liner fabrication technique was developed. When tested with liquid hydrogen the glass-flake liner was shown to be thermally compatible with the filament-wound shell. However, no conclusion could be made concerning strain compatibility because the tank could not be tested at a pressure greater than 1 atmosphere due to leaks.

INTRODUCTION

It is known that the use of hydrogen as the propellant in either a chemical or nuclear rocket results in a significantly higher specific impulse than any other fuel. Unfortunately, when in a liquid state, hydrogen has some undesirable properties that make storage difficult and thereby seriously limits its performance as a propellant. Since the liquid has a very low density (4.4 lb/ft^3) larger and hence heavier fuel tanks are required. This increases the need for an ultralightweight tank structure.

As a part of a general research program on liquid-hydrogen tankage problems, pressurization, insulation, and structures, the NASA Lewis Research Center is investigating the use of fiber-glass reinforced plastics as a structural material for tanks. Fiber glass has a distinct weight advantage over stainless steel or titanium. In fact, filament-wound motor cases for solid-fuel rockets have been used successfully with considerable savings in weight.

Although fiber-glass tanks are lightweight, they have a serious limitation. Because of their nonhomogeneous nature, filament-wound tanks leak. Therefore an internal liner or sealer is required. The liner must be capable of high strain since the ultimate strain of glass fiber is high (3 to 5 percent at room temperatures). This is no problem at room temperatures, but at liquid-hydrogen temperatures (normal boiling point -423° F) the problem becomes very severe. At -423° F the ultimate strain of glass fiber remains

high (3 to 5 percent), but the ultimate strain of conventional liners and sealers is greatly reduced. Also, in general, the thermal contraction of most materials is much greater than that of glass fiber. The development, then, of a suitable sealer or liner is one of the major problems associated with the use of fiber glass as a structural material for liquid-hydrogen tanks.

The investigation to date has been mainly experimental in nature, involving the actual fabrication and testing of small scale tanks with liquid hydrogen, since theoretical analysis has been hampered by the lack of knowledge of materials properties. These investigations are described in references 1 to 5.

This report is concerned with one possible method of lining a fiber-glass tank, that of using a liner made of several layers of small, overlapping flakes of glass, interspersed in a resin and oriented parallel to the wall of the fiber-glass shell.

The objectives of the research reported herein were to design, fabricate, and test in liquid hydrogen, a small-scale tank having the following specifications:

(1) An outer shell constructed by the filament-winding process

(2) An internal liner made of glass flake

(3) An integral fill and vent end fitting made of fiber-glass-reinforced plastic

This program was also undertaken to evaluate the feasibility of such a tank configuration.

The tank was fabricated by Owens-Corning Fiberglas Corporation, Granville, Ohio, for NASA Lewis Research Center.

The details of the design and fabrication along with experimental test results are presented in this report.

GENERAL LINER DESIGN CONSIDERATIONS

A criterion for liner integrity (derived in refs. 2 or 3) is:

$$\epsilon(P) < (1 - \nu)\epsilon_{ult} - (S_L - S_S) \tag{1}$$

where $\epsilon(P)$ is the tank strain due to pressure, ν is Poisson's ratio for the liner material at -423° F, ϵ_{ult} is the ultimate uniaxial liner strain at -423° F, and S_L and S_S are the unrestrained thermal contractions from room temperature to -423° F, of the liner and shell, respectively.

This expression assumes Hooke's law for the liner material to hold to the ultimate, a liner that has neglible thickness in comparison to the fiber-glass shell, the dimensions of the liner (before cooling) to be the same as the inside dimensions of the fiber-glass shell, the maximum principal stress theory of failure (ref. 6) and a fiber-glass shell of balanced design; that is, all the filaments carry the same load upon pressurization.

(The more general case of an unbalanced design is covered in refs. 2 and 3.)

The severity of the liner problem is emphasized by the data shown in table I. Given are some typical material properties for fiber-glass-resin composites and proposed liner materials. The column on the right is the quantity $(1 - \nu)\epsilon_{ult} - (S_L - S_S)$, which corresponds to the value of tank strain $\epsilon(P)$ at which the liner will fail (eq. (1)). The best of the shown plastics can only allow the fiber-glass shell to develop about one third of its ultimate tensile strain. The negative number for Teflon indicates that a small pressure (that which is necessary to expand the liner to the inside dimensions of the shell after cooling to - 423° F) will rupture the liner.

Some metals, on the other hand, have high ultimate tensile strains at -423° F (see table I). It would seem conceivable that a liner could be made out of a thin metal foil or a laminate of foils. The problem is, however, that the high metal strain is mostly inelastic. That is, the metal permanently stretches or yields. Upon tank pressurization, then, the proposed metal liner yields. When the pressure is released, the liner, which is now oversized, tends to debond from the tank wall, fold and wrinkle. Metal foils in general have a poor resistance to wrinkling and will develop pin-hole leaks.

Since the present day materials are inadequate, the liner problem can be solved only by advancing the state of the art. To do this, at least one of the following basic approaches to the problem must be employed:

(1) Do an extensive materials study to find a suitable liner material that satisfies inequality (1). Since there is probably no off-the-shelf material that meets these requirements, a suitable material would have to be created.

(2) Use a metal foil liner, but develop an adhesive bond that is so strong that there would be no chance that the liner could separate from the tank wall. The liner would then yield inelastically in both tension and compression. The fact that fiber-glass-reinforced plastic composites tend to crack or craze may cause difficulties with this approach. The metal liner would tend to become debonded at a crack in the shell resin.

(3) Allow the liner to wrinkle, but find ways of controlling the wrinkling processes so that pin holes will not develop. This wrinkling process involves simultaneous folding of the liner in two directions which is further complicated by the internal tank pressure. Before a successful liner can be developed using this approach, the wrinkling processes should be studied and thoroughly understood. In addition, if plastic films or laminates are used, a way of building extra material into the liner must be employed since the ultimate strain is limited (table I).

(4) Allow the liner to leak as long as the leak rate is below an acceptable limit. The liner is so constructed that when it cracks, the escaping fluid is forced to follow a labyrinth-type path. To accomplish this, a material in the form of flat plates, shingles, or tape is interspersed in a relatively weak resin matrix (see fig. 1). When a crack in the liner forms, it cannot propagate across any of the flat plates since they are of a much

stronger material. Since the width of the crack parallel to the flat plates or shingles is small and the path of the crack is long, the leak rate through the crack is small.

The investigation reported herein concerns itself with the last of these approaches wherein the flat plates or shingles are glass flakes of irregular shape.

APPARATUS AND PROCEDURE

Shell Design and Fabrication

The procedure followed in the fabrication of the small-scale (18-in. -diam, 24-in. -long) test tank used in this investigation was basically the same used for full-sized tanks and rocket motor cases, except for the glass-flake liner. This procedure is described in reference 10.

The test tank is composed of the liner, the end fittings, and the filament-wound shell. Details of the design of the mandrel, the end fittings, and the shell are presented in the appendix.

Development of the Glass-Flake Liner

Flakes of "E" glass (ref. 10), 0.00015-inch thick and 1/8- to 1/2-inch across were used. The main problems in incorporating these into a liner were:

(1) Obtaining a uniform distribution of glass flakes

(2) Trying to make the ratio of glass flake to resin as high as possible

(3) Orienting the glass flakes parallel to each other

(4) Applying the flakes gently so that they will not get broken into smaller pieces

Several methods of flake application were evaluated by applying the flakes to a small $5\frac{3}{4}$-inch-diameter mandrel. A thin fiber-glass cylinder (about 6-in. long) was wound over the liner. The flake liner was then tested by cold shocking the cylinder in liquid nitrogen. As a result of this, the following method of fabrication was developed:

(1) Flakes of uniform size were used and were those that passed through a 1/2-inch mesh screen and not through a 1/8-inch mesh screen.

(2) A thin coat of resin was sprayed on the mandrel using a Bink's Model 62 spray gun.

(3) Then a layer of glass flake was applied using a flocking gun.

(4) The above method of alternately applying resin and flake was continued until several layers of flake were built up.

For the first trial tank, two glass-flake liners were used; one on the inside of the

vessel and the other half way through the helical wrap. Each liner was composed of three layers of flake.

After completion, the tank failed to pass a prescribed preliminary test which consisted of the following:

(1) Pressurize the tank to 25 pounds per square inch gage at room temperature and leak check using a soap film as a detecting agent

(2) Thermally shock using liquid nitrogen

(3) Repeat test step (1)

The first tank had many small leaks on the initial pressurization to 25 pounds per square inch gage. However, few if any additional leaks developed after the thermal shock. Unsuccessful attempts were made to patch the leaks.

The reason this liner failed was thought to be caused by one or more of the following:

(1) Slippage of the flakes when winding the fiber-glass shell over them

(2) The mold-release agent working into the inner glass-flake liner

(3) Not enough layers of glass flake in either of the liners

(4) Voids or air bubbles in the liner

A second tank was then fabricated with the following changes:

(1) Only one liner (on the inside of the tank) was used.

(2) Six layers of glass flake were used in the liner.

(3) Each glass-flake layer was rolled to force out air and excess resin and to insure proper orientation of the flakes.

(4) The liner was B-staged (partially cured) before winding the fiber-glass shell to prevent slippage of the glass flake.

(5) No mold release was used. (This resulted in small chunks of the plaster, used to patch the mandrel, sticking to the inside of the tank.)

Figure 2 is a photomicrograph of the cross section of the wall of the second tank. The section was made perpendicular to the circumferential filament wrap (little white circular dots in the bottom half of the picture). The helical wrap appears as ovals. The lines in the top part of the picture are the glass flakes in the liner. The dark areas are voids.

The second tank was subjected to the same preliminary test as the first tank. Unfortunately the second tank received a bruise before the test. The bruised area, which was visible in the glass-flake liner, was sealed by applying glass flake and resin to the interior at the bruised spot and glass flake, resin, and a mat of glass fibers to the exterior. Upon the initial pressurization to 25 pounds per square inch gage, the tank was found not to leak. After the thermal shock with liquid nitrogen the tank was found to have two defective areas that leaked. These areas were sealed in the same manner as the bruised spot. The tank was then repressurized and found not to leak. It was thus considered ready for testing with liquid hydrogen.

Liquid-Hydrogen Test Facility and Procedure

Facility. - Figure 3 shows the facility that was used for testing with liquid hydrogen. The tank was mounted in a bell jar which had a two-fold purpose. First, the vacuum in the bell jar insulated the tank so that it could be kept full of liquid hydrogen for a reasonable length of time. Secondly, the bell jar provided a fixed volume so that the leak rate of the tank could be determined by rate of change of pressure in the bell jar.

In general, one of the major problems encountered when determining leak rate by measuring pressure change in a fixed volume is the elimination of leaks from sources other than the object of interest. The problem is further aggravated by cryogenic temperatures. The hardware used in this test was designed so that all low-temperature vacuum seals were vacuum protected. That is, at least a partial vacuum is maintained on both sides of a seal to eliminate the pressure driving force causing the leak. This was accomplished by placing the bell jar within another vacuum chamber (see fig. 3), and a small groove (in which a vacuum was continually drawn) within the flanged joint connecting the fiber-glass test tank to the stainless-steel vent line.

The instrumentation (also shown in fig. 3) consisted of:

(1) A pressure transducer of the strain-gage type

(2) A capacitance-type liquid-level probe

(3) Hot-filament-type vacuum gages used to determine the leak rate of the tank

Test procedure. - The testing of the tank is divided into three sequential steps:

(1) The initial leak rate is established, using helium at room temperature in the tank, for the purpose of evaluating the condition of the tank before filling with liquid hydrogen.

(2) The tank is thermally cycled at a constant internal pressure of 1 atmosphere. One thermal cycle involves first cooling the tank to -423° F with liquid hydrogen and then warming the tank to room temperature using warm helium. The ability of the liner to withstand repeated thermal stresses is evaluated by examining the leak rate.

(3) The tank is pressure cycled after filling with liquid hydrogen and attaining thermal equilibrium. As will be discussed later, it was not possible to perform this step.

Method of determining leak rates. - For determining small leak rates the bell-jar valve (shown in fig. 3) is kept closed. The leak rate L corrected to standard conditions of temperature and pressure is then given by the simple expression:

$$L = KV_1 \frac{T_S}{T_1} \frac{dP_1}{dt}$$

where: P_1, V_1, and T_1 are the pressure, volume, and temperature of the bell jar;

T_S is 460° R; t is time and K is a unit conversion constant, 1.04×10^{-5} (std. cm^3/sec)/(μ ft^3/hr).

For large leak rates, the bell jar must be pumped upon continuously in order to maintain a reasonable vacuum. With the bell-jar valve open, then, the preceding expression must be modified to:

$$L = KV_1 \frac{T_S}{T_1} \frac{dP_1}{dt} + KV_2 \frac{T_S}{T_2} \frac{dP_2}{dt} + \frac{T_S}{T_2} W(P_2) - E$$

where in addition P_2, V_2, and T_2 are the pressure, volume, and temperature of the pump manifold; $W(P_2)$ = pump rate as a function of P_2; and E = extraneous leaks into the pump manifold (assumed constant).

RESULTS AND DISCUSSION

The initial helium leak rate was found to be 2.18×10^{-2} cubic centimeter per second with helium at 1 atmosphere pressure and room temperature in the tank. Upon filling with liquid hydrogen at atmospheric pressure the leak rate increased to an unacceptably large value (shown in fig. 4). With further thermal cycling the leak rate leveled off indicating no further damage to the liner. Although, in figure 4, the room temperature helium leak rate and the hydrogen leak rate at -423° F are different, when corrected to standard condition, the volumetric leak rates at their respective temperatures are nearly the same. Therefore the size of the hole or leak path was not affected by temperature. Due to the high leak rate, it was impossible to test the tank at internal pressures above atmospheric which is unfortunate since this is the most important test of a liner.

Upon examination of the tank after the test, the largest leak was found to be from one of the previously mentioned patched areas located on the upper dome near the neck fitting. Two much smaller leaks were also found which did not originate from any patched areas.

Examination of the liner showed no signs of crazing, cracking, or delamination which indicates that the glass-flake liner is at least thermally compatible with the filament-wound shell.

CONCLUDING REMARKS

In this preliminary investigation a possible technique was developed for fabricating a glass-flake liner for a filament-wound fiber-glass liquid-hydrogen tank.

This liner was shown to be at least thermally compatible with the filament-wound

shell. Unfortunately, however, no conclusion can be made regarding the liner's strain compatibility since the tank used in this investigation could not be tested with liquid hydrogen at a pressure higher than 1 atmosphere.

The fact that the glass-flake liner is thermally compatible with the filament-wound shell suggests some possible uses for the glass flake. For example, assume that a resin was developed that was compatible in strain with a filament-wound shell. Resins often have the undesirable characteristics of having a high porosity and high thermal contractions (which leads to thermal stress which could cause a large thin-walled vessel to buckle). Glass flake introduced into such a resin would reduce its porosity and at the same time would eliminate buckling problems by reducing thermal stresses.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, September 21, 1965.

APPENDIX A

DESIGN OF FILAMENT-WOUND TANK

Mandrel

The mandrel was made of salt (NaCl) blocks, glued together and machined to the correct shape. Holes and cracks that developed during machining were filled with plaster. The mandrel was then covered with a compound (Dow-Corning DC-4 mold release) to prevent the resin used in the filament-winding processes from sticking to the plaster.

Filament-Wound Shell

The shell was wound using 12 strand, S-HTS Fiberglas roving and Bakelite ERLA 2256 epoxy resin with ZZ LB 0820 amine hardener. A conventional winding pattern consisting of a helical and circumferential wrap was used. The winding angle (angle between a fiber-glass strand and the longitudinal axis of the tank) of the helical wrap was 20.3°. The dome contour of the ends of the shell was of a standard geodesic-isotensoid type (ref. 11). The strand densities (in the cylindrical portion of the shell) were approximately 428 strands per inch for the helical wrap and 682 strands per inch for the circumferential wrap. One strand is a bundle of 204 individual filaments and has a cross sectional area of glass of 2.08×10^{-5} square inch. The above densities are the average of measurements made at three locations in the shell wall. The measurements were made by making actual end counts from photomicrographs (fig. 2 is an example) of the tank cross section.

Knowing the strand density and thickness of each wrap (0.019 and 0.032 inch for the helical and circumferential wraps, respectively), the glass content (by volume) of the shell can be calculated:

Helical wrap

$$G_h = \frac{D_h A}{T_h} = \frac{(428)(2.08\times10^{-5})}{(0.019)} = 47 \text{ percent}$$

Circumferential wrap

$$G_c = \frac{D_c A}{T_c} = \frac{(682)(2.08\times10^{-5})}{(0.032)} = 44 \text{ percent}$$

where

G glass content by volume

T thickness of wrap

D strand density

A cross-sectional area of glass in one strand

The other 53 and 56 percent of the helical and circumferential wrap, respectively, are air and resin (see the voids shown in fig. 2). The reason for the smaller glass content for the circumferential wrap is that there is a resin buildup on the outer surface of the tank.

Also knowing the strand density, the strength of the fiber-glass shell can be calculated. Using the method outlined in reference 2 the relation between shell strain and pressure (shown in fig. 5) can be calculated. The ultimate strain of "S" glass (ref. 10) is about 5 percent. However, when glass fiber is used in a filament-wound vessel, the ultimate strain of the glass is reduced due to imperfect packing of the helical fibers as they cross over each other in the vessel ends and imperfections such as variation in resin content, broken fibers due to handling, nonuniform winding tension, and nonuniform strand distribution. A realistic ultimate shell strain is about 3 percent (ref. 10). From figure 5, this corresponds to a burst pressure of about 600 psi.

End Fittings

At each end of the tank (at the center of the domes) is a fitting. At one end (bottom) the fitting is simply a plug. At the other end the fitting (neck fitting), through which the tank can be filled and vented, is used to attach the tank to the liquid-hydrogen test facility.

To eliminate thermal stresses and thus minimize the chances of a leak, the end fittings were made of the same material as the filament-wound shell.

The neck fitting was a filament-wound 4.0-inch-diameter cylinder with a 1/4-inch wall thickness (fig. 6). The cylinder was made up of several layers of helical windings at an angle of 60° to the axis, and one layer of circumferential windings.

The flanges were made by drawing up the ends of the filament-wound cylinder and forming them by pressing the filaments and uncured resin between molds or forming tools of the correct shape.

The bottom plug was made by first winding several 4-inch-diameter by 1/16-inch-thick cylinders. The uncured cylinders were cut and pressed into flat plates. Several disks cut from the flat plate were stacked and pressed between molds or forming tools to give a 3/8-inch thickness and the correct shape as shown in figure 6.

The uncured or partially cured fittings were then fitted over the glass-flake liner on the mandrel followed by the winding of the shell. The fittings were then cured at the same time as the rest of the tank to insure a good bond.

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TABLE I. - ULTIMATE STRAIN AT -423° F AND THERMAL CONTRACTION FOR SOME MATERIALS

Material	Ultimate tensile strain, ϵ_{ult}, in. /in.	Overall thermal contraction 75° to -423° F S_L, in. /in.	Reference	Maximum allowable tank strain, $\epsilon(P)$, in. /in. (calculated) (a)
Fiber-glass-resin composite	[b] 2.7×10^{-2}	1.8×10^{-3}	2	--------
Mylar "A" film	0.8×10^{-2}	3.8×10^{-3}	5	0.4×10^{-2}
"H" film	2.0	5.3	5	1.15
Polyurethane	2.0	16.3	5	.05
Teflon, FEP	2.0	22.0	7	-.02
2024 Aluminum	20.0	4.2	8, 5	(c)
304 Stainless steel	30.0	3.0	8, 9	(c)

[a] Calculated by equation (1). This involves ν (Poisson's ratio) which is unknown for these materials at -423° F. Here, ν was assumed to be 0.25, a theoretical value for an ideal isotropic material.

[b] This number should probably be higher since all test specimens failed in jaws of apparatus.

[c] Maximum $\epsilon(P)$ could not be calculated for these materials from relation presented in text since these materials strain mainly by yielding.

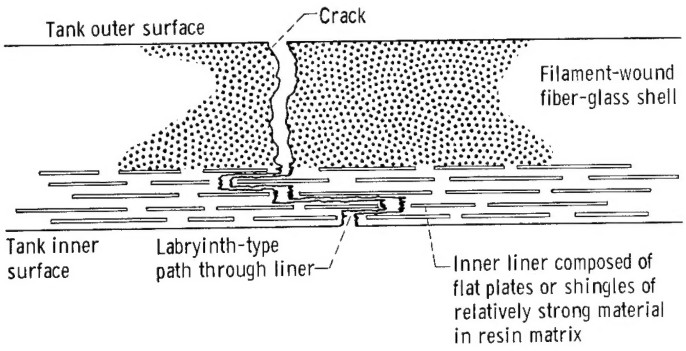

Figure 1. - Principle of flake- or shingle-type liner.

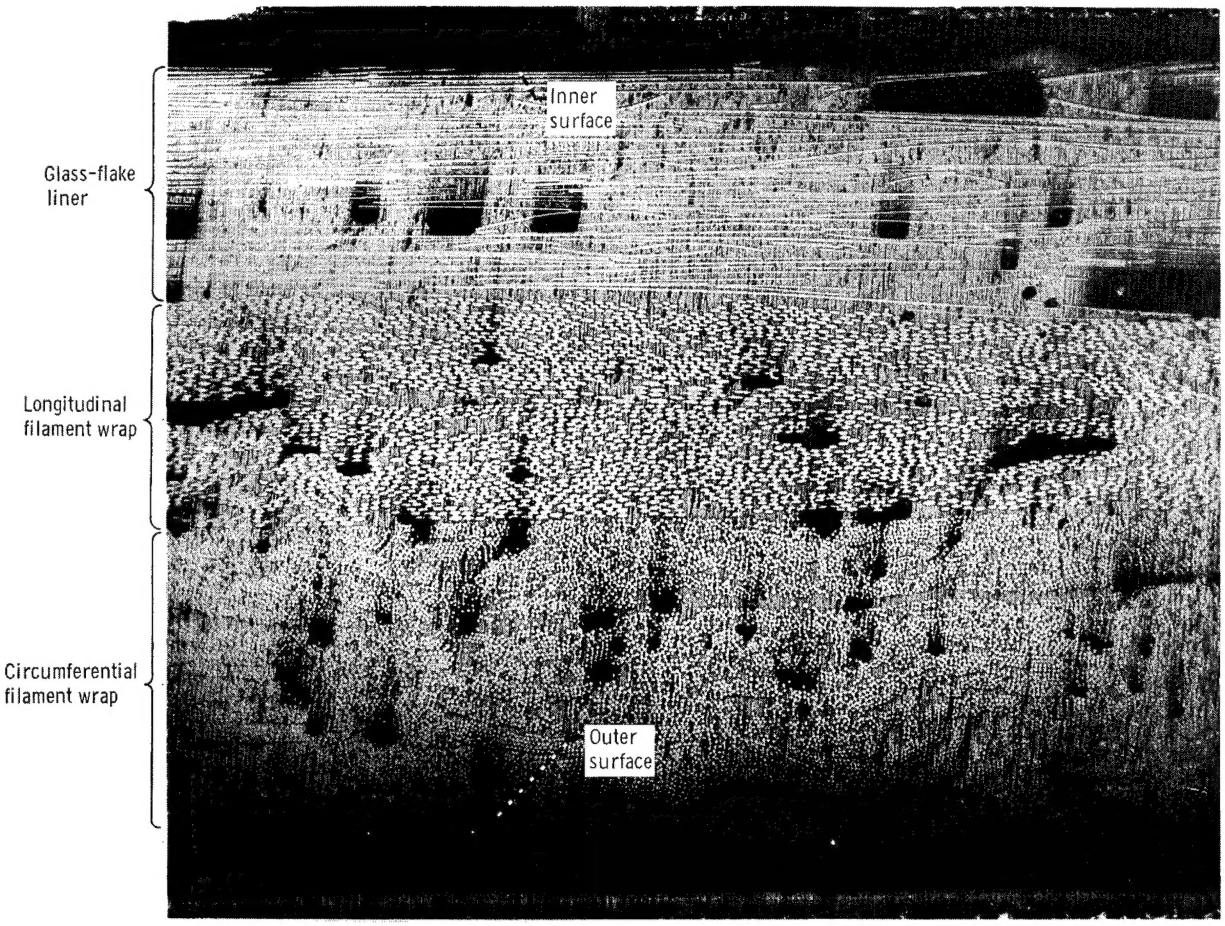

Figure 2. - Cross section of filament-wound tank with glass-flake liner.

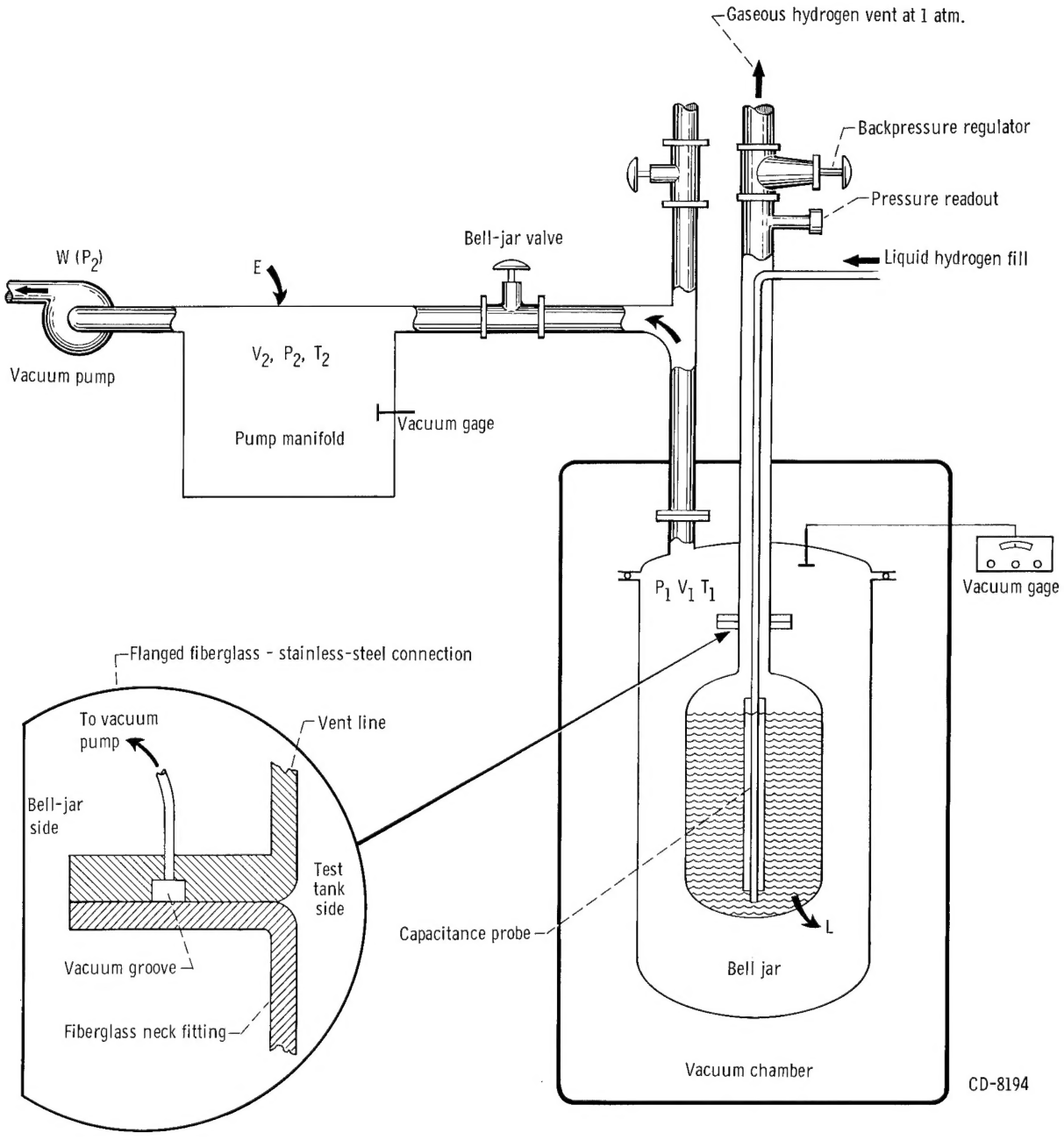

Figure 3. - Liquid-hydrogen test facility.

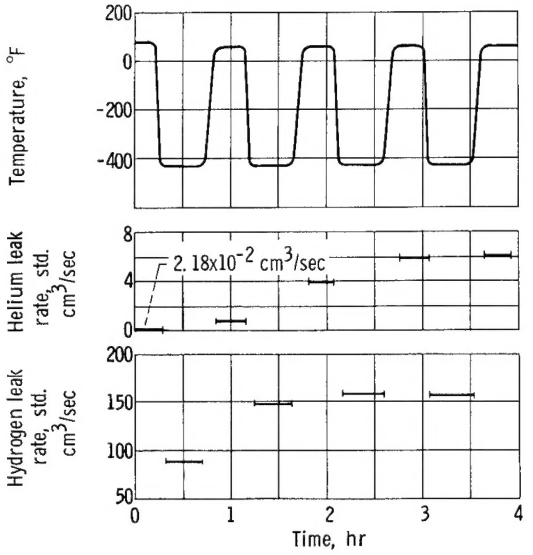

Figure 4. - Leak rate while thermal cycling with liquid hydrogen at 1 atmosphere pressure.

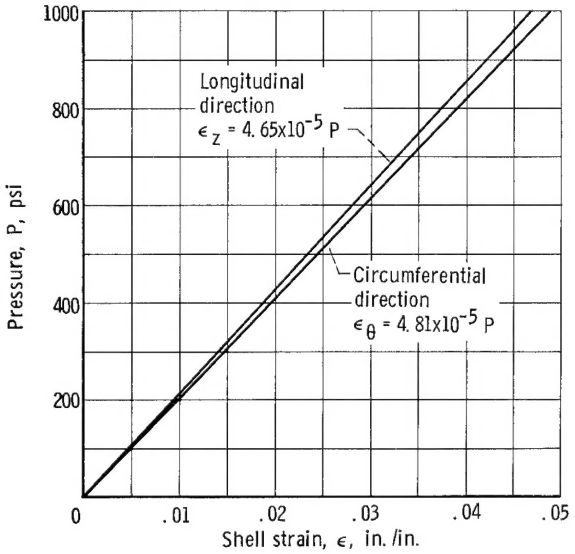

Figure 5. - Calculated shell strain as function of pressure at ambient temperatures.

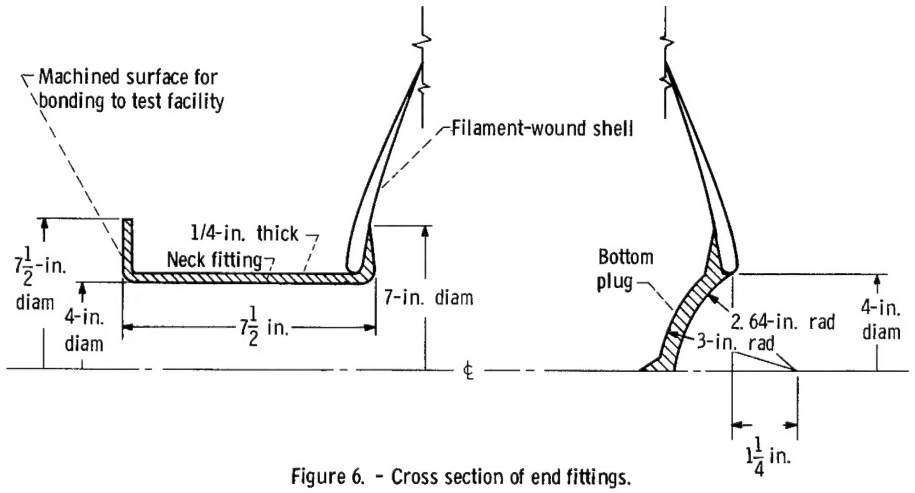

Figure 6. - Cross section of end fittings.

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